

PATENT
CASE 4233C3

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Donald B. Appleby et al : Group Art Unit: 1211

Serial No.: 08/360,184 : Examiner: E. White

Filed: December 20, 1994 :

For: **Polyol Polyester Synthesis**

DECLARATION UNDER 37 C.F.R. §1.608 OF PAMELA D. HARDY

Assistant Commissioner for Patents
Washington, DC 20231

Dear Sir:

I, PAMELA D. HARDY, declare that:

1. I am employed by the assignee of the present application, The Procter & Gamble Company, and have been working for The Procter & Gamble Company continuously since 1974.
2. I received a Bachelor of Arts Degree from Edgecliff College in 1977.
3. From 1987 to 1998 I worked as an analytical technician on the sucrose polyester synthesis project in which sucrose and fatty acid methyl ester were reacted to form sucrose fatty

acid esters. I was responsible for analyzing samples of reactor streams from pilot plant continuous sucrose polyester production processes according to established procedures.

4. I participated in the pilot plant continuous sucrose polyester production process designated P90117 which was conducted from January 18, 1989, to January 27, 1989. The P90117 pilot plant continuous sucrose polyester production process, including operation of the continuous sucrose polyester synthesis equipment, sample collection and data preparation, was performed under the direction and control of Mr. Scott Pearson.

5. During the P90117 pilot plant continuous sucrose polyester production process, I determined the average degree of sucrose esterification (based on mole fractions and referred to as the I-bar value) and the area percentages of monoester, diester, triester, tetraester, pentaester, hexaester, heptaester and octaester, respectively, in the sucrose ester, in samples taken from the recirculation loops of the reactors according to established procedures described below. I accurately recorded the determined average degree of sucrose esterification (I-bar) and area percentages of monoester, diester, triester, tetraester, pentaester, hexaester, heptaester and octaester, respectively, in the sucrose ester, in the samples on data sheets which were maintained in the pilot plant for the purpose of recording such data. The data which I accurately recorded included the date and time at which a sample was taken, the reactor recirculation loop from which the sample was taken, and the determined average degree of sucrose esterification (I-bar) and area percentages of monoester, diester, triester, tetraester, pentaester, hexaester, heptaester

and octaester, respectively, in the sucrose ester, in the sample. I also initialed the entry for each sample which I analyzed.

6. I have examined Exhibit 8, and I confirm that Exhibit 8 comprises accurate copies of the data sheets from the P90117 pilot plant continuous sucrose polyester process into which I accurately entered the date and time at which a sample was taken, the reactor recirculation loop from which the sample was taken, and the determined average degree of sucrose esterification (I-bar) and area percentages of monoester, diester, triester, tetraester, pentaester, hexaester, heptaester and octaester, respectively, in the sucrose ester, in the sample. The entries in Exhibit 8 under the heading "SFC" represent area percentages determined using supercritical fluid chromatography according to the established procedures described in the following ¶7. The entries in Exhibit 8 under the heading "HPLC" represent area percentages determined using high performance liquid chromatography according to the established procedures described in the following ¶8. Generally, SFC was used during the P90117 pilot plant continuous process to determine area percentages of sucrose esters in samples from reactors R600, R601 and R602 while HPLC was used during the P90117 pilot plant continuous process to determine area percentages of sucrose esters in samples from reactors R603, R604, R605 and R606. The entries bearing my initials (PH) indicate those samples which I analyzed as described herein and for which I recorded the data as described herein.

7. The established procedure for determining the area percentages of monoester, diester, triester, tetraester, pentaester, hexaester, heptaester and octaester, respectively, in the

sucrose ester, in a sample by supercritical fluid chromatography (SFC) was as follows: Each reactor sample was silylated to derivatize any unreacted hydroxyl groups and the silylated sample was injected into a supercritical fluid chromatograph. In the chromatograph, the esters were separated by degree of esterification on a capillary column and detected by a flame ionization detector. The detector output comprised a graph containing a series of peaks representing, respectively, the distribution of individual sucrose esters, on an area basis, in the sample. The area percentages of the individual sucrose esters relative to the total sucrose ester content in a reactor sample were determined using the equation:

$$\% \text{ SE}_n = \frac{\text{Area SE}_n}{\sum \text{Area SE}} \times 100$$

wherein n indicates the number of ester groups on a sucrose ester, Area SE_n is the area under the peak corresponding to the "n" ester and \sum Area SE is the sum of all of the areas under the ester peaks.

8. The established procedure for determining the respective area percentages of the sucrose esters by high performance liquid chromatography (HPLC) was as follows: Each reactor sample was dissolved in hexane, and the resulting solution was filtered and then injected into a high performance liquid chromatograph equipped with a silica column. The mobile phase for the chromatography was a methyl-t-butyl/hexane step gradient system. The respective sucrose ester area percentage in the sucrose ester sample was calculated by the chromatograph integrator based on output from the chromatograph light-scattering mass detector.

9. The I-bar was calculated on a mole fraction basis from the area percentages of monoester, diester, triester, tetraester, pentaester, hexaester, heptaester and octaester determined by chromatography, assuming the area percentages of the respective sucrose esters are substantially equivalent to weight percentages, according to the equation:

$$I\text{-bar} = \frac{\sum_{n=1}^8 n \times \underline{\text{Area \% n ester}}}{\sum_{n=1}^8 \frac{\underline{\text{Area \% n ester}}}{\text{Mol. Wt. n ester}}}$$

wherein "Mol. Wt." is the molecular weight of the respective ester.

10. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the present application or any patent issued thereon.

Respectfully submitted,

By: 
Pamela D. Hardy
Date: 3/31/99

0350682.05